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Pragmatic approach to cure profile enhancement for improved fatigue performance of thermoset matrix composites

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Abstract. The current paper proposes a low cost scheme for enhancement of the fatigue life properties of fibre reinforced composites through minimization of residual stresses induced from the composite cure cycle. The enhancement scheme works on the presumptions that a low processing temperature at the event of resin gelation causes reduced residual stresses. The requirements for material characterization and numerical implementation of the scheme is low compared to optimization schemes available in the literature. The enhancement scheme is implemented and used to produce enhanced two stage cure cycles for a commercially available epoxy resin.

1. Introduction

Most of the fibre reinforced composites manufactured today are based on thermoset matrix resins which intrinsically builds up residual stresses in the final composite products. Recent studies have found that high levels of residual stresses impair the fatigue life properties of fibre reinforced composites. Hüther et al [1] showed that the fatigue properties of UD non-crimp fabric composites were impaired by cure cycles with higher amount of process induced strains. These findings were supported by Mikkelsen et al [2]. Mikkelsen et al measured the magnitude of process induced strain due to various temperature profiles in neat resin samples and used the same temperature profile when curing otherwise identical composite laminates reinforced with non-crimp glass fibre fabrics. Mikkelsen et al [2] found that the composite laminates cured with temperature profile that produced high magnitudes of process induced in the neat resin samples had impaired fatigue life properties compared composite laminates cured with temperature profiles that induced low amounts of strain in the neat resin samples. Warnet et. al [3] conducted bending experiments on cross-ply laminates and observed a decrease in experimental energy release rate of bending specimens when the residual stresses where increased. Experimental observations on transverse cracking in cross-ply laminates were also made recently by Zobeiry et al [4]. In their work laminates were produced with two different cure cycles that produced different levels of residual stresses in the cross-ply laminate. It was shown that higher levels of residual stresses caused transverse matrix cracks to form at lower levels of applied strains during mechanical testing. Asp et al [5] came up with a possible reason for the residual stresses impair the strength of composites. Asp et al showed that cavitation-induced cracks could occur before matrix yielding in polymers where significant hydrostatic stresses existed.

Cure profiles of composites can be designed to minimize the residual stresses in the final laminate. A key observations was made by Prasatya et al [6] who concluded that a direct relation existed between the temperature of the resin material at the point of gelation and the resulting residual stresses in the final composite material. The gelation point were described as the point where the resin material changes from liquid to solid phase. The observations made by Prasatya is supported by a recent study made by Mortensen et al [7] in which an optical Fibre Bragg Grating (FBG), made from silica, was embedded in a neat resin sample during cure. Mortensen et al found that tempeature at the point of gelation resulted in lower strains induced to the FBG fibre. Other researches have made conclusions that implicitly supports the idea of that low temperatures of gelation. Genidy et al [8] concluded that curing resins at a low temperature for a long time increased effective thermal expansion [when heating from the first to second temeperature stage of a two stage cure, red], because it counteracts the effect of chemical and cool down shrinkage. White and Hahn [9] conducted experiments on unsymmetrical laminates to find an optimized cure profile for lower residual stresses and concluded that lowering the initial cure temperature and holding it for longer time reduced the residual stresses while maintaining the mechanical properties. They noted that laminates with degrees of cures less than 0.95 had inferior properties to the full cured laminates, but that laminates with a higher than 0.95 degree of cure performed as well as laminates cured with the manufacturer recommended cure cycle. White and Hahn [9] also noted that high magnitudes of cool down rates reduced the residual stresses build up in the composite laminates. Gopal et al [10] focused on the cooling and heating rates of two stage cures, and claimed that for a two stage cure cycle it was optimal to go from the first temperature hold to the second temperature hold as fast as possible. Shah et al [11] made a complex constraint optimization of cure cycle parameters to reduce residual stresses. The results for two stage cures showed that the temperature of the first hold was generally lowered when the cure was allowed to take up more time. While Shah et al did not point this out them selves, the tendency of their optimized cure cycles fit with the idea of minimizing the temperature of gelation in order to minimize residual stresses. The work of Shah et al [11] succeeded in producing an optimization scheme that can effectively lower the magnitude of residual stresses, but the sheer complexity of proposed method makes it unsuitable for industrial purposes. The method requires implementation of very sophisticated numerical methods and expensive experimental material characterization of the desired resin for manufacturing.

The following work will present a scheme for designing two stage cure cycles for thermoset resin based composites with minimized levels of residual stresses based on a 0-D numerical cure model that is suitable for thin laminates. The scheme requires relatively inexpensive material characterization and a relatively low effort in terms of implementation into a numerical code. The idea behind the scheme is to design two-stage cure cycles that minimizes the temperature of gelation as a method for minimizing the resulting residual stresses. The scheme will be subject to a constraint of maximum allowable manufacturing time, as well as an additional constraint of material quality based on the thermoset resin degree of cure.

2. Cure kinetic model and Process simulation

The degree of cure of a thermoset resin is often defined by the amount of reaction heat released pr. unit mass of the material H , relative to the ultimate amount of reaction heat pr. unit mass H_U [12] [13]. This definition is expressed in eq. (1). The rate of the resin cure follows from the same definition, thus being proportional to the rate of heat of reaction as prescribed in eq. (2). Dynamic (temperature ramp) DSC measurement are widely used to determine the ultimate heat of reaction H_U . Figure 1 shows three measurements performed on the resin used in this article where the crosshatched area for each of the heat flow curves represents the ultimate heat of reaction H_U for each of the measurements. Isothermal DSC scans, as shown in Figure 2, are used for non-linear regression to determine the reaction order model.

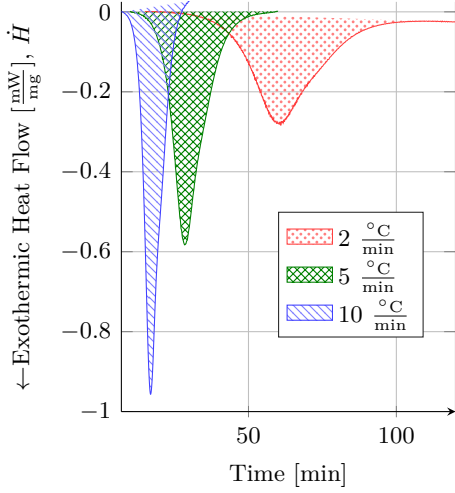


Figure 1: Dynamic DSC measurement of an epoxy resin with different heating rates. All measurements were started at -20 °C and ended at 300 °C.

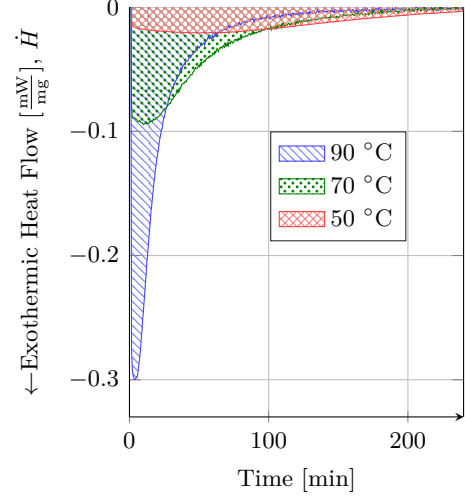


Figure 2: Isothermal DSC measurement of an epoxy resin. Each measurement were kept at the isothermal temperatures for 4 hours.

$$\alpha(t) = \frac{H(t)}{H_U}, \quad \alpha \in [0; 1] \quad (1)$$

$$\frac{d\alpha}{dt} = \frac{1}{H_U} \frac{dH(t)}{dt} \quad (2)$$

The general form of most phenomenological cure kinetic models can be described by eq. (3) where the term $f(\alpha)$ is the reaction model which is specific to the applied cure kinetics relations. The $K(T)$ term on the right hand side is the temperature dependence of the reaction rate, which, for all cure models described in the current paper, will be defined by an Arrhenius expression as printed in eq. (4). The constant A [-] is a pre-exponential constant, E [$\frac{\text{J}}{\text{mol}}$] is the activation energy, T [K] is the material temperature and $R = 8.3145$ [$\frac{\text{J}}{\text{K}\cdot\text{mol}}$] is the ideal gas constant.

$$\frac{d\alpha}{dt} = K(T) f(\alpha) \quad (3)$$

$$K = A \exp\left(-\frac{E}{RT}\right) \quad (4)$$

$$f(\alpha) = \alpha^m (1 - \alpha)^n \quad (5)$$

The cure kinetic model for thermoset resins that will be applied in the current work is classified as a phenomenological model. The expression for the reaction model $f(\alpha)$ is printed in eq. (5). The reaction order parameters n and m as well as the reaction rate constants of the Arrhenius expression A and E are determined using non-linear regression. The data input for the non-linear regression model is the degree of cure, computed using eq. (1), and the rate of cure, computed using eq. (2), as well as the corresponding temperature. Figure 3 shows the non-linear regression data fit to each of the isothermal DSC measurements. The degree of cure and rate of cure in Figure 3 is derived from the isothermal measurement data shown in Figure 2. The table in Figure 3 presents the parameters found for the cure kinetic model used in the regression, and these parameters along with the phenomenological cure model of eqs. (3)-(5) will

be applied in section 5. A more detailed description of the process of obtaining the parameters for the cure kinetic model can also be found in Khoun et. al [14] or Yousefi et al. [12].

Having established a cure kinetic model, and the corresponding parameters for the model, simulation of the resin cure can be performed using numerical integration tools such as MatLab's ODE solver¹ or SciPy's odeint² library or other numerical tools. Solving the cure kinetic equations with a given initial value for the degree of cure will be denoted as a cure simulation in section 4 of this paper.

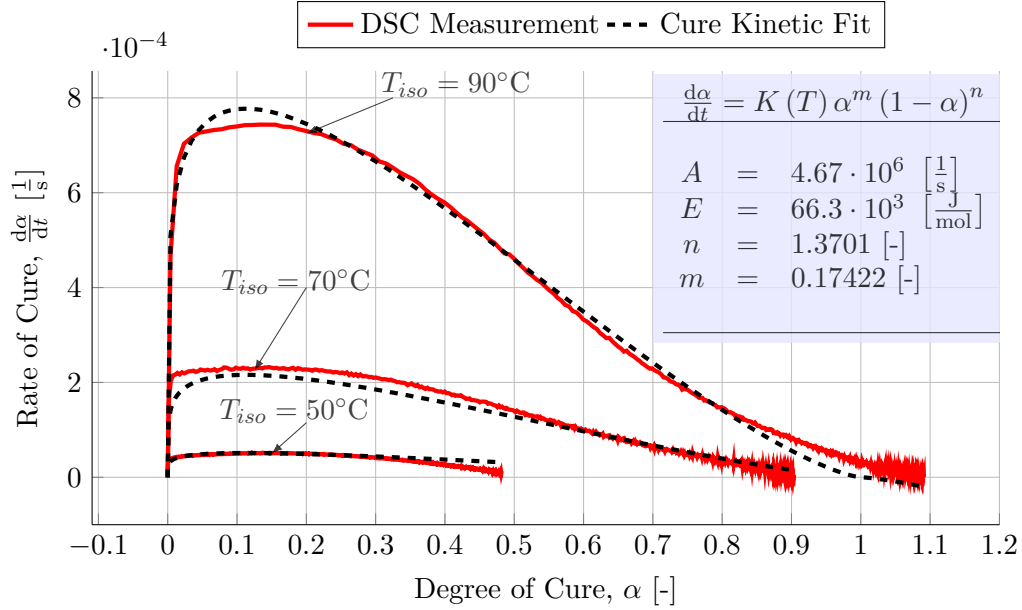


Figure 3: Fit of cure kinetic model (eq. (5) with input from the isothermal DSC measurements.

3. Gelation and Process Induced Strain

While at least one study [4] have suggested that tool-part interaction can produce internal stresses when the thermoset matrix is in the liquid phase it is generally assumed in the literature that no significant stresses can arise prior to gelation (see e.g. [13] [14] [15] [16]). The residual stresses σ_{res} in a thermoset matrix composite is then dependent on the cure history *after* gelation and the mechanical constraints dictated by the method of manufacturing. The point of gelation is most often found using rheometry as this technique readily spans both the liquid and solid phase of polymeric materials and can be used to generate visco-elastic material models [14]. Khoun et al used rheometry to establish a degree of cure of gelation of $\alpha_{gel} = 0.7$ [14]. From the perspective of polymer chemistry the point of gelation can be defined as the time of critical conversion (degree of cure) where infinite three-dimensional polymer networks can exist [17]. This definition allows the gelation degree of cure to be computed based on stoichiometry of the resin. O'Brien [13] did however note that the theoretically calculated degree of cure of gelation from the resin they investigated was found to be $\alpha_{gel} = 0.58$, significantly lower than the point they obtained using rheometry which was $\alpha_{gel} = 0.71$. Khoun et al [14] and O'Brien [13] both defined the gelation as the point of equality between the storage and loss moduli of

¹ <https://se.mathworks.com/help/matlab/numerical-integration-and-differential-equations.html>

² <https://docs.scipy.org/doc/scipy/reference/generated/scipy.integrate.odeint.html#scipy.integrate.odeint>

their respective rheometry measurements.

3.1. Temperature of Gelation

In a recent study by Mortensen et. al [7] the total process induced strain was found to be closely related to the temperature of gelation T_{gel} . The study measured chemical and thermal strain using optical Fibre Bragg Gratings (FBG) on a neat epoxy sample that was unaffected by mechanical constraints. The phenomenon that is thought to govern the relation between temperature of gelation and total process induced strain can be illustrated by the the strain development during cures shown in Figures 4 and 5 for a one stage and a two stage cure respectively. It should be noted here that the event of gelation is assumed to occur at a specific degree of cure, α_{gel} the degree of cure of gelation, independently of the material temperature at the instant of the gelation. This means that the temperature of gelation T_{gel} is defined as the temperature of the material in the instant that the degree of cure of gelation α_{gel} is reached such that $\alpha(T, t_{gel}) = \alpha_{gel}$. The gelation of a resin will be defined as the point in time where the resin can obtain normal forces and are capable of inducing measurable deformations of small objects, such as a single glass fibre FBG. Furthermore, the total process induced strain at room temperature ε_{tot} is assumed to be the sum of the chemical (shrinkage) strain ε_{ch} and the thermally induced strain ε_{th} as given by eq. (6).

$$\varepsilon_{tot} = \varepsilon_{ch} + \varepsilon_{th} \quad (6)$$

The measurements shown in Figures 4 and 5 are based on the experiments conducted by Mortensen et al [7]. In the one stage cure shown in Figure 4 the gelation point are reached when the temperature is close to the isothermal cure temperature. The temperature after gelation is nearly constant such that no thermal strains are expected to develop during the temperature hold. The strain induced during the temperature hold (region A1 in Figure 4) are caused solely by chemical shrinkage of the resin. When the temperature hold ends and the material is cooled and compressive thermal stresses arise due to thermal contraction of the material. This means that using a one stage cure produces a total process induced strain that is the sum of the total amount of chemical shrinkage strain and the thermal strain of the temperature difference from the cure temperature to the operating temperature, which in most cases are at room temperature of the final manufactured part.

For the two stage cure shown in Figure 5 the point of gelation occurs at the end of the first temperature hold, thus causing a low temperature of gelation. After the material has reached gelation, and is capable of attaining normal stresses, the processing and material temperature are increased. In region A2 in Figure 5 the temperature is increased. The temperature increase causes the the rate of cure to increase, thus resulting in chemical strain that causes compression. The chemical strain is however counteracted by thermal expansion of the resin, resulting in a net positive increase in strain. In the A2 region in Figure 5 the thermal expansion and chemical shrinkage acts in opposite direction. After the peak, when the material temperature is constant, only chemical strain due to curing are acting on the FBG strain sensor, causing a slight decrease in strain. At the end of the second temperature stage the process induced strain is in tension before the material is cooled to room temperature where only thermal strains are induced to cause a final state of compression. The final level of total process induced strain is lower for the two stage cure due to the thermal expansion during the temperature ramp up that happens after gelation and counteracts the chemical shrinkage. The level of process induced strain before the final cool down shows an important difference between the one stage and two stage cures because it must be assumed that the final cool down to room temperature must cause similar amount of thermal stresses for both of the materials. In the on stage cure the strain before the final cool down equals the inevitably level of chemical strain, while for the two stage the

strain is in the tension regime before the final cool down. It should further be remarked that a significant porting of the the thermally induced strain comes from the final cool down of the material to room temperature, as pointed out in region C2 of Figure 5, and as such the maximum temperature of the second temperature stage should be identical to the ultimate T_g^{ult} of the resin material such that full cure can be achieved with as low a second stage temperature as possible.

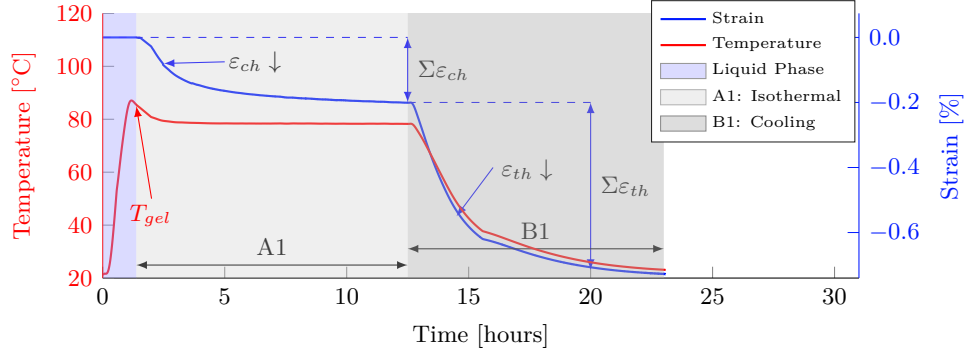


Figure 4: Strain and Temperature development over time for a one stage cure with 80°C curing temperature for 12 hours.

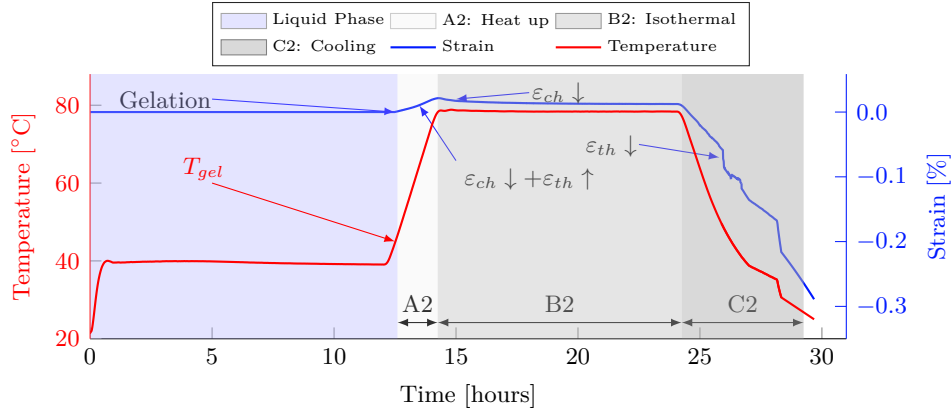


Figure 5: Strain and Temperature development over time for a two stage cure with 40°C for 12 hours followed by 80°C for 12 hours.

The experiments conducted by Mortensen et al [7] also included implicit measurement of the degree of cure using dielectric analysis. This allowed the degree of cure of gelation to be established as $\alpha_{gel} = 0.66 \pm 0.05$ for the epoxy resin. This value, $\alpha_{gel} = 0.66$, will be used in cure simulations in section 5.

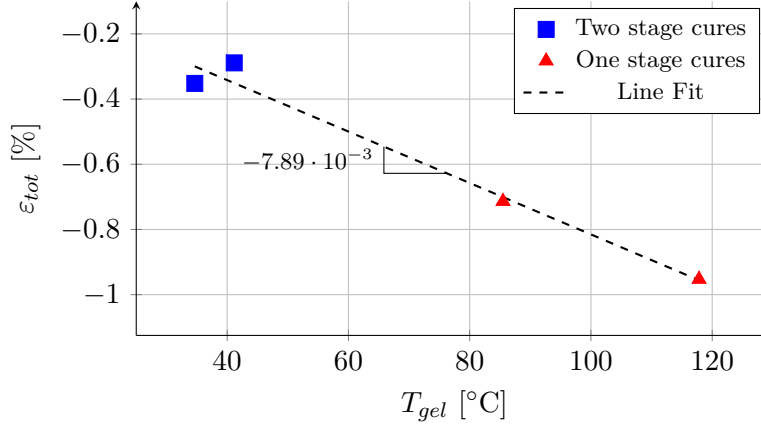


Figure 6: The total process induced strain at room temperature (25 °C) plotted against the temperature of gelation T_{gel} .

Figure 6 shows how the total process induced strain is related to the temperature of gelation for the epoxy used by Mortensen et al [7]. The two data points with low temperatures of gelation is a product of two stage cures, and the high temperature of gelation results from one stage cures. There is a clear tendency for the total process induced strain to be increasingly compressive (negative) for an increase in the temperature of gelation. This tendency constitutes an important observation when designing cure cycles with the aim of lowering the residual stresses: Even though the measured process induced strain in a neat epoxy sample are not directly proportional to the level of residual stresses in a manufactured composite, the clear negative correlation between temperature of gelation and process induced strain means that the residual stresses in a composite must also increase in magnitude for an increase in the temperature of gelation. The validity of this principle is also supported in the literature where Prasatya concluded that *"the results demonstrate a direct relationship between the temperature at which gelation occurs and the magnitude of isotropic residual stresses at room temperature"* [6].

4. Cure optimization

4.1. Problem formulation

Based on the works of Hüther et al [1], Warnet et al [3] and Mikkelsen et al [2] an increased level of residual stresses σ_{res} in thermoset composites leads to impaired fatigue life of the composites. An improved cure cycle for a composite must then minimize residual stresses in order to improve the fatigue life of a thermoset matrix composite. An overall goal of minimizing the residual stresses can be posed as a constraint minimization problem, as printed in eq. (7), where the objective function is the total process induced strain subjected constraints of maximum time t_{max} , maximum temperature T_{max} and minimum attained degree of cure α_{end} . The optimization problem does however come with the caveat of having an output variable (α_{end}) as non-linear constraint. Furthermore, having process induced stresses, or strains, as the objective function requires expensive experimental resin characterization in order to build a thermo-chemical viscoelastic material model.

$$\begin{aligned}
& \mathbf{min} && \sigma_{res}(T, t) \\
& \mathbf{subject\ to} && \max(t) \leq t_{max} \\
& && \max(T) \leq 1.2 \cdot T_g \\
& && \alpha(T, t = t_{max}) \geq \alpha_{end}
\end{aligned} \tag{7}$$

Instead of solving the non-linear constraint optimization problem posed in eq. (7) the observations from section 3 can be applied to simplify the problem. Specifically the consequence that minimizing the temperature of gelation T_{gel} also minimizes the total process induced strains, and hence the residual stresses, is applicable in formulating a more suitable minimization problem. The revised problem, for an assumed two stage cure, is given in eq. (8). It should be noted that the constraints are the same as given in eq. (7), but temperature of gelation T_{gel} can not readily be defined as an objective function.

$$\begin{aligned}
& \mathbf{min} && T_{gel} \\
& \mathbf{subject\ to} && \max(t) \leq t_{max} \\
& && \max(T) \leq 1.2 \cdot T_g \\
& && \alpha(T, t = t_{max}) \geq \alpha_{end}
\end{aligned} \tag{8}$$

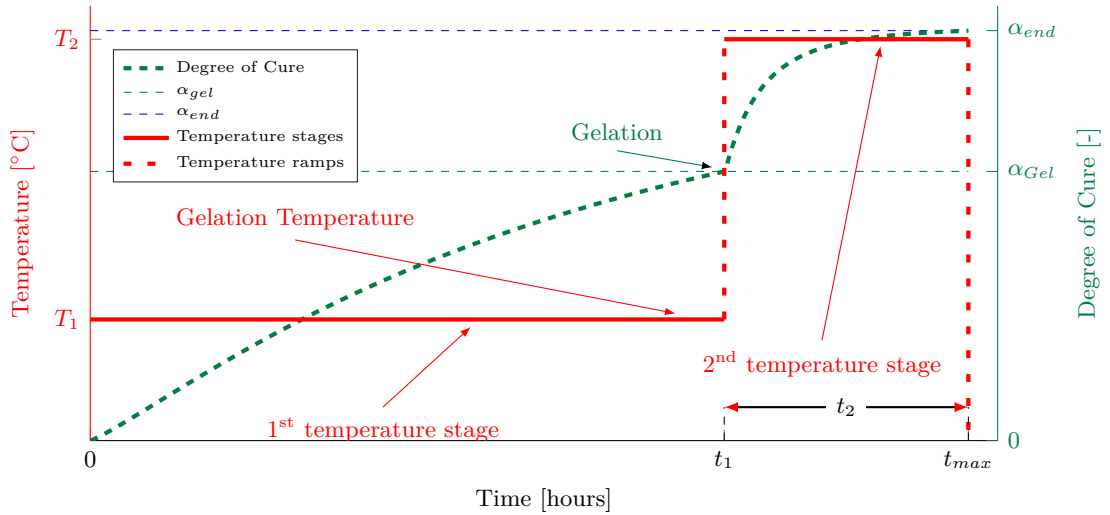


Figure 7: Principal sketch of a the parameters that define the optimal two stage cure.

4.2. Minimization of the Temperature of Gelation

The solution to eq. (8) will not be defined here in terms of classical optimization, but will instead apply some known quantities about the resin cure cycle aimed to be enhanced. Figure 7 shows schemtically the parameters that define a two stage cure cycle, excluding heating and cooling ramps to and from the temperature stages. The temperature of the first stage T_1 and second stage T_2 and the amount of time, t_1 and t_2 respectively, the temperatures are held at

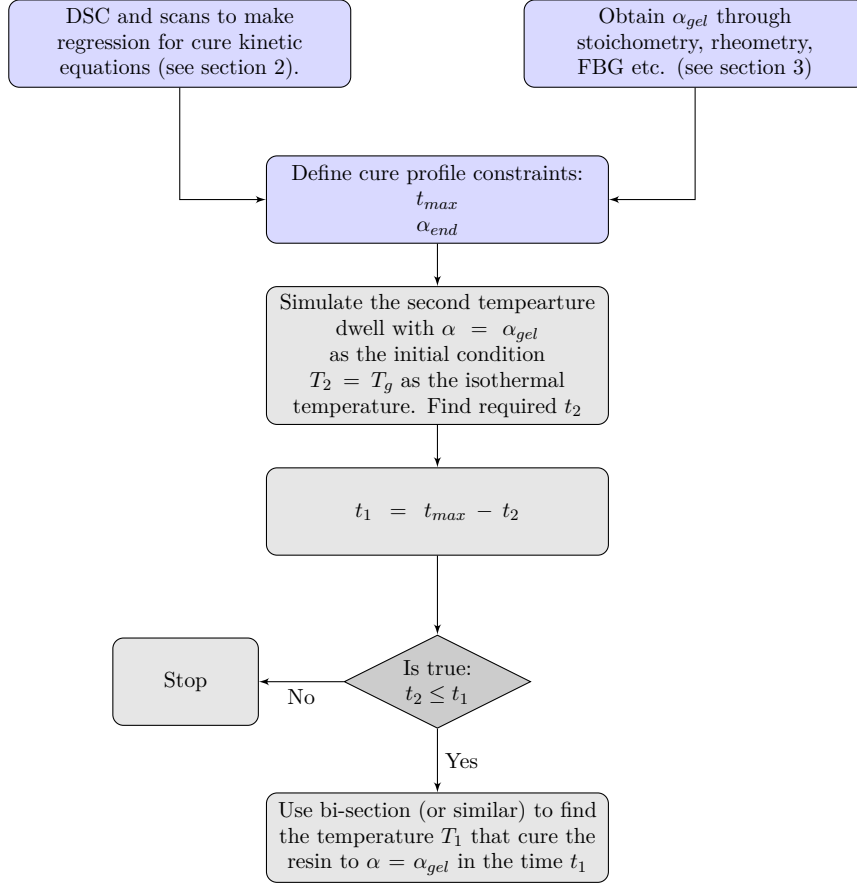


Figure 8: Flowchart of the minimization of the temperature of gelation. Blue boxes are experimental procedures and user input. Grey boxes are simulation process steps.

those stages. The following will assume that the degree of cure of gelation α_{gel} is a known resin quantity and that the temperature T_2 of the second stage is identical to the ultimate T_g of the resin material, henceforth denoted T_g^{ult} . The choice of $T_2 = T_g^{ult}$ for the second curing stage is based on the general need for the process temperature of the material to reach the temperature of ultimate glass transition temperature to ensure cure. In other words T_g^{ult} is the lower bound temperature the resin needs to reach in order to attain a full cure. The development of the degree of cure can be simulated by numerical integration of the cure kinetics equation from section 2. Using $\alpha = \alpha_{gel}$ as initial condition and $T = T_2 = T_g^{ult}$ the time (t_2) required for the resin degree of cure to reach the specified minimum degree of cure α_{end} , can be found. When t_2 is a known time the amount of time (t_1) available for the first temperature stage is known as the remaining time from the defined maximum available time t_{max} . The required solution to eq. (8) can then be reduced to finding the temperature T_1 of the first temperature stage that satisfies eq. (9).

$$\alpha(T_1, t_1) = \alpha_{gel} \quad (9)$$

The described procedure for minimizing the temperature of gelation T_{gel} is schematically outlined in Figure 8. An additional condition are advised in the flowchart in Figure 8, namely the requirement that the time for cure during post-gelation t_2 should not exceed the time t_1 for cure during pre-gelation. The condition is based on practical experiments from simulations that shows the process to be ineffective if $t_2 > t_1$.

5. Results

The optimization process described in section 4 has been implemented in a MatLab routine. The routine follows the simulation steps (grey boxes) in Figure 8 and takes in α_{end} and t_{max} as constraint parameters. The routine furthermore takes a cure kinetic function and a set of parameters as arguments as well as a value for the gelation degree of cure. For the results shown in this section applies the cure kinetic model is described in section 2, and the specific model fit and parameters that are shown in Figure 3. Figure 9 shows the found cure cycles with three common time constraints of 8, 16 and 24 hours. All three of the shown cure cycles were subjected to the constraint of $\alpha_{end} \geq 0.99$. The decreasing of the initial temperature T_1 resulting for increase of the maximum allowable time should come as no surprise; the entire optimization flow was designed to do exactly this.

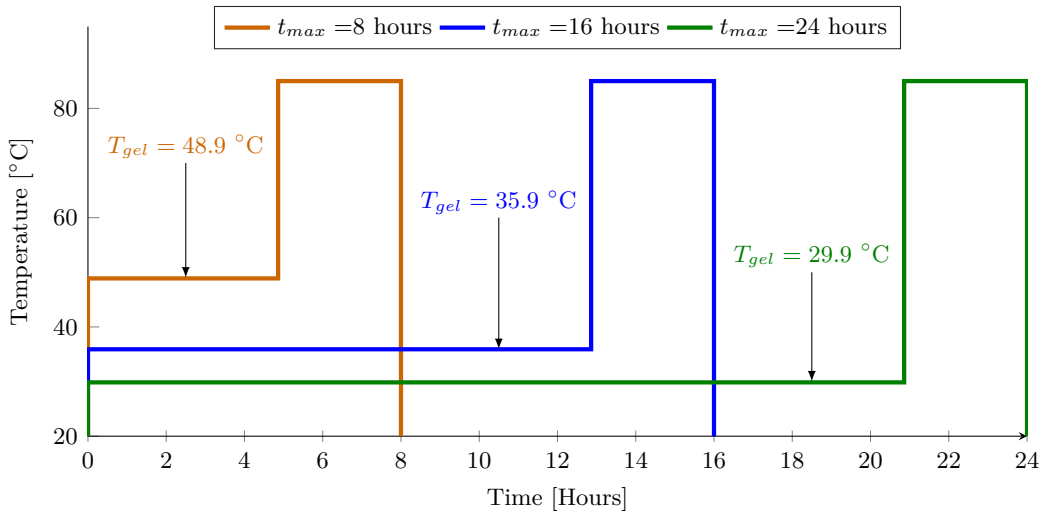


Figure 9: Cures with minimized temperature of gel

Given that the temperature of gelation found using the optimization process is a direct product of the time and curing constraints, t_{max} and α_{end} respectively, a small investigation into the effect of the constraints were conducted. Figure 10 shows the temperature of gelation, found using the optimization process, plotted against the time constraint t_{max} . The Figure shows curves for various levels of the constraint for the minimal attained degree of cure α_{end} . Each of the curves in Figure 10 starts at the value of t_{max} that allows at least half of the cure time to be spent at the low curing temperature t_1 . This essentially means that the first point of each of the curves are the lowest t_{max} value where the $t_2 \leq t_1$ requirement is fulfilled. The resulting curves shows that having a tight requirement of the minimum attained degree of cure makes the optimization less effective, and essentially means that longer time is needed to reach a degree of cure that satisfies the constraint. Figure 10 also shows that significant decreases in the temperature of gelation can be achieved by increasing the the maximum allowed time for the curing of a composite laminate. Especially cure cycles constrained to take less than 5 hours could see significant lowering of the temperature of gelation by allowing additional time for curing.

6. Discussion

The implementation of the proposed cure profile optimization have been implemented and tested on a commercially available low temperature epoxy. The applied degree of cure of gelation in the results was $\alpha_{gel} = 0.66$. This was the mean value report in the experiments by Mortensen

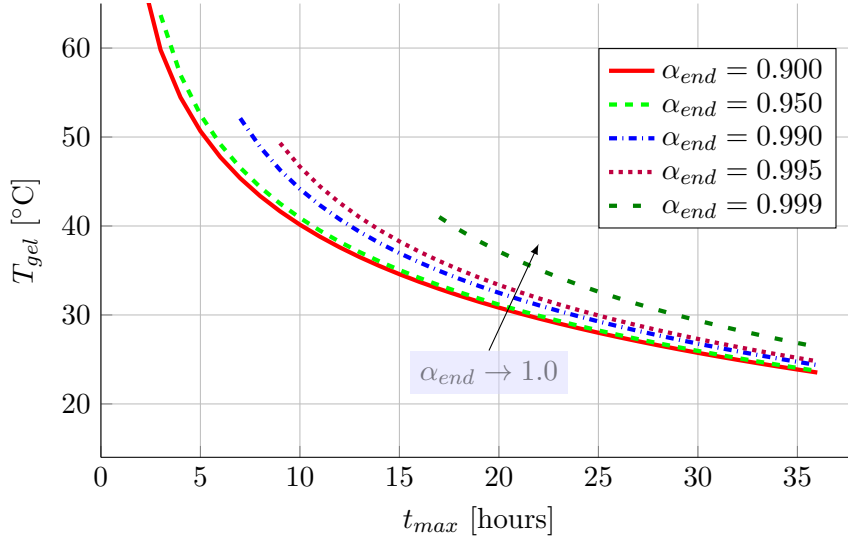


Figure 10: The obtained value for the temperature of gelation plotted as an output function of the time constraint t_{max} . Various curves for the minimum attained degree of cure α_{end} are shown.

et al, who also reported an uncertainty of ± 0.05 . It may be argued that since the optimization scheme will only function if the α_{gel} is reached at the low temperature stage the proper α_{gel} input value to the optimization would be $\alpha_{gel} = 0.66 + 0.05 = 0.71$. In general it should be suggested that the α_{gel} input should be the upper bound value of experimental observations of α_{gel} . Common values of α_{gel} for epoxies are 0.6 - 0.7 [9] [13]. Other types of thermoset resins may have significantly different values for α_{gel} . Polyesters are reported to have gelation degree of cures as low as $\alpha_{gel} = 0.1 - 0.3$, which may also mean that the prospect of changing the temperature of gelation of a cure cycle for other types of thermoset resins are very different than the prospect for epoxy resins.

The requirement for the minimum acceptable degree of cure α_{end} after completing the curing cycle may also affect the outcome of the optimization process. It is noted here that White and Hahn [9] showed that epoxies cured to less than 0.95 degree of cure suffered from impaired mechanical properties. It is thus recommend that $\alpha_{end} \geq 0.95$ for epoxies.

The optimization process outlined in this paper is based on a 0-D curing model that do not take into account the thermal conduction problems of laminates with finite thickness. For thin plates with thickness's less than 3 mm the through thickness thermal gradients can be assumed to be low enough that the cure model will provide sufficiently accurate results. For thick laminates the cure kinetic model would have to be implemented into a 1-D thermo-chemical model in order to account for temperature and cure gradients through the laminate thickness. The procedure would have to satisfy eq. (9) at all points through the thickness of the laminate. The procedure would also have to avoid high exothermic reactions during the first temperature stage. Low exothermic peaks are however an intrinsic property of low temperature cures as the low cure rate guarantees a low exothermic heat release rate. The optimization process also excludes the required temperature ramp both from the room temperature to the first temperature stage T_1 as well as the temperature ramp from the first to the second temperature stage with temperature T_2 . In order to implement a temperature cycle in a practical application these temperature ramps can of course not be neglected. The temperature ramps can be included either by using a fixed heating rate and deducting the time needed to heat from room temperature to T_2 , which will correspond to the total time needed for heating. More sophisticated approaches would include additional iterative procedures and will not be further detailed here.

7. Conclusion

A scheme for optimizing thermoset resin cure cycles has been presented. The scheme takes advantage the idea that lowering the temperature of gelation for a thermoset resin will result in lower magnitudes of residual stresses in composite laminates. The outlined optimization scheme requires a low degree of experimental characterization because it does not use on residual stresses or strains as an objective function, which would require costly resin characterization experiments in order create a cure dependent thermo-viscoelastic material model. Instead the optimization relies on minimizing the temperature of gelation of the thermoset resin used in composite manufacturing. The use of the temperature of gelation also means that the implementation of the scheme in a numerical code is greatly simplified compared to the required numerical implementation of cure dependent visco-elastic material models in a classical constraint optimization scheme.

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